

FEB 16 2006**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

Applicant: Steve Sak-kyoun Ow and Tae Jin Eom

Serial No.: 09/121,152 Art Unit: 1731

Filed: July 22, 1998 Examiner: Anna Kinney

For: *BIOLOGICAL DEINKING METHOD*

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R. § 1.132

Sir:

I, Howard Kaplan, hereby declare that:

1. I am employed at Ezymatic Deinking Technologies, Norcross, GA, as its chief operating officer. Enzymatic Deinking Technologies is the licensee of the above-identified patent application.
2. I instructed my laboratory manager, Jian Hua Ma, to conduct experiments to compare the deinking of recycled paper using the conditions described in Example 2 of Japanese Patent Application No. 59-9299 ("JP '299") and the above-identified application.
3. I reviewed JP '299 to determine the conditions and materials described therein for the enzyme enhanced deinking of recycled paper. The only conditions were described in the examples. Example 1 added a number of materials other than an enzyme and NaOH. Example 2 examined the effect of adding 1% by weight NaOH and an alkaline cellulase. It was my

U.S.S.N. 09/121,152

Filed: July 22, 1998

DECLARATION UNDER 37 C.F.R. § 1.132

understanding that the examiner preferred we use the conditions of Example 2 so that there would be fewer variables. We therefore conducted a comparison of the deinking of recycled paper as described in example 2, with the claimed method which requires a pH of less than 8, differing in the pH of the reaction mixtures and the cellulases which were added. Each experiment was performed 10 times to provide a statistically valid result. The results of the experiments are enclosed.

4. Example 2 does not provide a pH of the reaction mixture but instead refers to adding 1% (relative to the old newspaper) NaOH. The average pH of the mixture after caustic addition was 11.19. The average pH of the mixture after disintegration was 11.12. The average pH of the mixture after addition of the enzyme was 11.16 and the average pH of the mixture after stirring was 10.67. For purposes of comparison, NaOH was not added to the reaction mixture of the claimed method. The average pH of the reaction mixture after stirring was 7.5.

5. It was not possible to obtain any of the enzymes described at page 3 of the JPA. We contacted Amano Pharmaceutical Co. and tried to locate Ueda Kagaku, listed as the manufacturers. We also searched a number of catalogs and on the internet. Amano did not sell the named enzyme and Ueda appears to be out of business. We then obtained an equivalent alkaline cellulase from Meiji Seika, HEP-100, an alkaline cellulase which is active over a range of at least 4.0 to 10.0, with a pH optimum of 8.0. For purposes of comparison, a neutral cellulase was obtained from Novozymes, Novozym 342 produced by the fungus *Humicola insolens*, which has an optimum pH of between 6.5 and 7.5.

U.S.S.N. 09/121,152

Filed: July 22, 1998

DECLARATION UNDER 37 C.F.R. § 1.132

6. As described in Example 2 of JP '299, each reaction mixture contained old newspapers, cut in 2 x 5 cm pieces, fed into a laboratory disintegrator, water and, for the JP '299 study, 1.0% NaOH, relative to raw material old paper, and disintegration done at pulp concentration 5%, 40°C for 20 minutes. After disintegration, 0.2% enzyme relative to raw material old paper as described in example 2 was added to the mixture containing the 1% NaOH and an equivalent amount of enzyme added to the other reaction mixture, and stirring was done at 45°C for one hour. The pulped material was then concentrated to 15% pulp concentration, diluted to 1% by adding water, and filtered through a Buchner funnel. The paper in the funnel and the filtrate were then analyzed.

7. The whiteness of the treated pulp (L-value) and the whiteness of the removed liquid (L-value) were determined for paper and filtrates from both samples.

The results showed that the treatment at the lower pH was more effective than the treatment at the higher pH, in the presence of 1% NaOH.

	<u>Paper L-value</u>	<u>Filtrate L-value</u>
JPA sample with 1% NaOH	65.9%	60.0%
Ow sample at pH 7.2	68.1%	56.4%

8. Not only were the results superior without NaOH treatment, but the cost of the treatment in the absence of the NaOH is reduced since the price of NaOH, at the time the application was filed, was about \$400/ton. The absence of 1% NaOH in the claimed method

U.S.S.N. 09/121,152

Filed: July 22, 1998

DECLARATION UNDER 37 C.F.R. § 1.132

would create a savings of approximately \$4.00/ton at the time the application was filed, or approximately \$6.80/ton today (See the attached abstract which discloses the price of caustic soda from 1988-1991). Mills typically process eight hundred tons per day, for a cost savings at the time the application was filed of \$3200/day, and operate 350 days/year year, leading to a cost savings of \$1.12 million/year as of the time this application was filed, or \$1.9 million today.

9. The undersigned declares that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true and further that these statements are made with the knowledge that willful false statements are punishable by fine or imprisonment or both under 18 U.S.C. 1001, and that such willful false statements may jeopardize the validity of the above-identified patent application or any patent issuing thereon.

Date: _____

Howard Kaplan

45053653

4

EDT 101 CON
0931462

Effect of caustic and enzymes on pulp and filtrate whiteness with mixed ONP

12/8/2005

Caustic with cellulase enzymes												
Exp. #	1	2	3	4	5	6	7	8	9	10	Average	Standard Deviation
NaOH, %	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	0.0
Alkaline MEJU HEP100, %	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
pH water	7.15	7.22	7.23	7.19	7.20	7.14	7.21	7.23	7.10	7.19	7.19	0.05
pH paper + water	7.18	7.17	7.16	7.20	7.15	7.23	7.18	7.24	7.16	7.17	7.19	0.04
pH after caustic addition	11.22	11.23	11.22	11.24	11.20	11.18	11.05	11.14	11.16	11.19	11.19	0.06
pH after disintegration	11.15	11.12	11.15	11.14	11.10	11.08	11.04	11.07	11.12	11.12	11.12	0.05
pH w/enzymes addition	11.14	11.19	11.18	11.24	11.20	11.13	11.16	11.04	11.09	11.22	11.16	0.06
pH final after stirring	10.70	10.87	10.70	10.65	10.65	10.53	10.59	10.62	10.76	10.87	10.87	0.05
Pulp whiteness, %	66.00	68.01	68.23	65.88	65.99	65.62	65.43	65.90	66.21	65.47	66.87	0.28
Filtrate whiteness, %	60.80	60.35	60.60	59.94	60.10	61.00	58.13	59.23	59.60	62.10	60.99	1.17

Caustic with cellulase enzymes												
Exp. #	11	12	13	14	15	16	17	18	19	20	Average	Standard Deviation
Novozyme SP342, %	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
pH water	7.16	7.16	7.16	7.20	7.17	7.16	7.18	7.16	7.16	7.16	7.17	0.01
pH paper + water	7.08	7.13	7.18	7.12	7.20	7.08	7.13	7.08	7.13	7.13	7.12	0.04
pH after disintegration	7.39	7.35	7.43	7.39	7.32	7.43	7.39	7.43	7.38	7.38	7.39	0.04
pH w/enzymes addition	7.39	7.36	7.43	7.39	7.32	7.42	7.40	7.43	7.38	7.38	7.39	0.03
pH final after stirring	7.51	7.47	7.55	7.51	7.44	7.55	7.51	7.55	7.51	7.50	7.51	0.04
Pulp whiteness, %	68.10	67.73	68.43	67.95	68.12	67.88	68.04	67.76	68.34	68.13	68.05	0.23
Filtrate whiteness, %	57.46	56.90	65.08	64.84	56.52	57.23	56.23	55.78	56.72	56.35	56.35	1.24

Procedure:

This set of tests was conducted following the procedures in the Example 2 in JP-A-53-929.

Mixed ONP was shredded into 2x5 cm pieces, 100 g was fed into a laboratory disintegrator with water and sodium hydroxide, the disintegration was done at about 5.0%, 40C for 20 minutes. After disintegration, 0.2% (based ONP fiber) enzymes was added, and stirring was done at 45C for 1 hour. It was then concentrated to about 15% pulp consistency, diluted to 1% by adding water and pulp sheet was made in a Buchner funnel. When the pulp was concentrated, the removed liquid was kept at 6C for 12 hours, and 200 ml of supernatant was taken out and the L value was measured.

Comments:

The tests were performed with mixed ONP collected around Metro Atlanta area. In December 2005, which has high inherent whiteness than Asian ONP. The results from these tests shall be different from all previous tests in terms of whiteness gain and ink removal before and after treatment, however the relationship between whiteness and caustic, or at different pH shall be very similar.

1.0% NaOH shifted the pH from 7.1-7.2 to 11.2, and the final pH dropped slightly with NaOH due to fiber absorption of alkalinity; and the final pH increased without NaOH due to chemicals leaching from paperfibers to the suspension. Enzymes showed no impact on pH of the fiber suspension.

PULP AND PAPER
"Chemistry and
Chemical Technology
Third Edition-Revised and Enlarged

Third Edition, Volume I

Volumes in preparation—

II FIBER PREPARATION AND APPROACH FLOW
FIBER BONDING
SHEET FORMATION AND DRYING
NONWOVEN AND PAPERS FROM SPECIAL FIBERS
SYNTHETIC PAPERS AND PULPS
ENVIRONMENTAL CONTROL
MICROBIOLOGY

III DRY STRENGTH RESINS
NATURAL PRODUCTS FOR WET-END ADDITION
FILLING AND LOADING
INTERNAL SIZING
RETENTION CHEMISTRY
WET STRENGTH
DYING OF PAPER
SURFACE SIZING
PROPERTIES OF PAPER

IV PIGMENT COATING
PRINTING
REPROGRAPHY
LAMINATING
CORRUGATING
PAPER REINFORCEMENT BY POLYMER ADDITION
PAPER LAMINATES
AQUEOUS AND SOLVENT COATINGS
EXTRUSION AND HOT MELT COATINGS

*A WILEY-INTERSCIENCE
PUBLICATION*
JOHN WILEY & SONS
New York Chichester
Brisbane Toronto

DISPATCHED FROM
McMINNVILLE, OREGON

CONTRIBUTORS

Walter J. Bublitz, Ph.D.

Professor, Pulp and Paper—Department of Forest Products
 Oregon State University—Corvallis, Oregon

J. R. G. Bryce, Ph.D.

Manager, Pulp, Paper and Packaging Research
 Domtar, Inc.—Sennerville, Quebec, Canada

Warren F. Daniel

Pulp and Paper Consultant
 Hanover, New Hampshire

H. P. Dixson, Ph.D.

Technical Advisor to President
 Fox River Paper Company—Appleton, Wisconsin

A. J. Falton

Director, Systems Technology—Shartle and Pandia Divisions
 The Black Clawson Company—Middleton, Ohio

Wolfgang G. Glasser, Ph.D.

Associate Professor, Wood Chemistry—Department of
 Forest Products
 Virginia Polytechnic Institute and State University—Blacksburg, Virginia

Copyright © 1951, 1960, 1980 by John Wiley & Sons, Inc.
 All rights reserved. Published simultaneously in Canada.

Reproduction or translation of any part of this work
 beyond that permitted by Sections 107 or 108 of the
 1976 United States Copyright Act without the permission
 of the copyright owner is unlawful. Requests for
 permission or further information should be addressed to
 the Permissions Department, John Wiley & Sons, Inc.
 The Permissions Department, John Wiley & Sons, Inc.

Library of Congress Cataloging in Publication Data:

Main entry under title:

Pulp and Paper.

First-2d ed. written by J. P. Casey.
 "A Wiley-Interscience publication."
 Includes bibliographies and index.
 1. Paper making and trade. 2. Wood-pulp.
 L. Casey, James P., 1915-
 TS1105.C29 1979 676 79-13435
 ISBN 0-471-03175-3 (v. 1)

Printed in the United States of America

10 9 8 7 6 5 4 3 2 1

TABLE 4-67 GRADES OF WASTEPAPERS

Mixed wastepaper—contains various qualities of paper not limited as to type or fiber content. Prohibitive material cannot exceed 2% and throwouts not to exceed 10%. Mixed wastepaper is used in roofing and bituminous asphalt shingles, molded articles, center ply in multiply board for boxes, structural boxes, Probative material, center ply in multiply board for boxes, structural dry-wall and common low-cost board.

Corrugated Waste—contains double-lined kraft outer surfaces and a fluted medium center. Also includes double-lined corrugated cuttings, corrugated cuttings, new kraft corrugated cuttings, and used corrugated containers, or boxes. Probative material cannot exceed 1% and throwouts cannot exceed 5%. This grade is used in the production of 'luncheon, corrugating medium, dry-wall board, and roofing. It comprises the largest tonnage in the secondary-fiber field, with annual consumption well in excess of 5 million ton in the United States.

Direct Entry (also identified as pulp substitute)—consists of white paper having no printing, of reasonable uniform brightness, and of no prohibitive material. Throwouts may not exceed 0.5%. This grade is used instead of virgin, bleached pulp in fine papers and publication papers.

Drinking Grades—consists of papers having printing, color, or groundwood content that can be treated in a delinking process that will remove the color, printing ink, and impurities. Should contain no prohibitive material and throwouts may not exceed 0.25%. This grade is used in the production of fine papers, book paper, envelope, and all types of tissue consumer products.

News—consists of baled, sorted, fresh, dry newspaper—not sunburned, and news from magazines, white blanks, pressroom oversize, and paper (other than news), containing not more than the normal percentage of rotogravure and colored sections. Packing must be free from tar. No prohibitive material allowed. Throwouts may not exceed 0.25%.

Prohibitive Material—any material that by its presence in the bale in excess of the amount allowed will make the bale unsuitable or unusable for the grade specified. Any material that may be physically damaging to the equipment.

Throwouts—all papers that are processed or treated in such a manner as to make them unsuitable for consumption in the grade specified.

TABLE 4-68 WASTEPAPER CONSUMPTION: DAILY USAGE IN THE UNITED STATES AND DOLLAR VALUE

Wastepaper Grade	Tons Daily Consumption	\$ Value as Wastepaper	\$ Value as Pulp
Mixed waste	7,532	75,320	542,340
Corrugated waste	15,506	620,240	3,821,910
Direct entry	498	99,600	136,800
Drinking grades	7,291	383,260	1,691,280
No. 1 news	6,217	248,680	475,470
	37,044	1,577,120	6,867,800

568 PULPING

residues, such as cornstalks, 1133 castorstalks and jute-sticks, 1124 can be used as supplementary fibrous materials. Rye grass straw (*Lolium multiflorum Linn.*), which is available in abundant quantities in the Willamette Valley, Oregon, has been found suitable by Bublitz¹¹⁵ for use as a supplementary fibrous material for papermaking in conjunction with wood pulp. Various types of grasses such as Lemon grass (*Cymbopogon citratus*), 1156 Ulta grass (*Andropogon giganteus*), 1157 thatch grass (*Imperata cylindrica*), 1158 and Elephant grass (*Themeda cymbaria*)¹¹⁵⁹ have been found suitable for pulping and are used for paper and board manufacture. Papyrus (*Cyperus papyrus*), which grows on the banks of the river Nile in Sudan and Egypt, is known from ancient times as a paper-making raw material. The rind, separated from the stalk of papyrus, contains fibrous material comparable to delipted bagasse. Harvesting, collection, and handling of these fibrous materials require extensive study to make the pulp and paper producers interested in their utilization.

SECONDARY FIBER PULPING

A. J. PELTON

The Black Clawson Company
Middletown, Ohio

Secondary fiber pulping involves the repulping of wastepapers and paperboard. There are two basically different methods: (1) a purely mechanical system involving the use of pulpers, screens, and centrifugal separators and (2) a combination chemical and mechanical system in which chemicals are used in the pulping stage to remove ink and other contaminants. For many years secondary fiber pulping did not keep pace with the overall growth of the paper industry, but recent economic factors and environmental considerations have caused it to expand greatly. Secondary fiber is the second largest source of fiber for paper and paperboard in the United States, USA, and the percentage of reuse of fiber is greater in Europe and Japan than it is in the United States.

Grades of Wastepaper

The pricing structure of wastepaper in the United States is built around a large number of different grades, but the most common grades are divided into 112 classes, 112x shown in Table 4-67. The volume of wastepaper used on an average daily basis from each class in the United States and the value of each, as wastepaper and as finished pulp, is shown in Table 4-68.

Mixed wastepaper is a particularly difficult grade to repulp because of its high

TABLE 4-69 GRADES OF CYLINDER BOARD

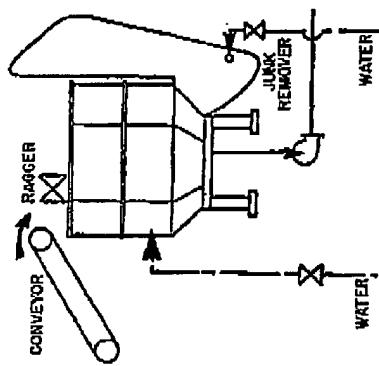


Figure 4-124. Conventional waste paper pulping system.

degree of contaminants, such as metal particles, stones, bottles, tapes, rags, strings and plastic materials, polystyrene, polyethylene, blown styrene, foamed styrene, hot melts, and bituminous asphalt. It requires a recovery system that will continuously remove these contaminants to a degree that allows paper to be made from the recovered fibers. It can be repulped by the system shown as Figure 4-124, which is used extensively in the United States. The pulping of mixed waste paper and corrugated waste is expected to increase in the future due to government pressure, economic conditions, tax incentives, and improved pulping systems. A substantial proportion of this increase will come from newly designed plants that will produce better, cleaner grades of secondary fiber for use in linerboard, corrugating medium, tissue, writing paper, and multiply board. The increased consumption of waste paper will result in a shortage and a deterioration in quality of the overall raw material supply. However, those mills that have the proper equipment, designed to clean and screen plastics and other light-weight contaminants, will be able to use this waste material without problems.

Multiple Cylinder Board

Much of the secondary fiber is used in multiply cylinder board, which is made on a cylinder machine, where the paper is built up to the desired thickness in separate layers. There are many grades of cylinder board and these are listed in Table 4-69. Secondary fibers are almost always used in the filler ply, whereas virgin pulp may be used in the outer (liner) ply. It is generally necessary to have separate pulp-preparation systems for the liner stock and the filler stock.

Secondary Fiber Pulping Systems

The mechanical systems used for the repulping of waste paper and paperboards are described in this section. There are a number of differently designed systems

Chipboard—Chipboard is a grade of cylinder machine board that contains the same stock in all plies and is always wastepaper stock. This board is made in a wide range of thicknesses and both "bending" and "nonbending" grades are made.

Bogus Bristols—Mills bristols, which are called by this name to distinguish them from index bristols made on a fourdrinier machine, are used mostly for poster cards and advertising. Bogus bristols are generally made from a number of different grades of stock, such as mixed waste, news, blank news, kraft waste, or virgin pulps. They are used for cheap cards, tickets, and colored tickets.

Folding-Box Board—If bending qualities are necessary, as in the case of folding-box boards, the two outside plies of the board must be made from long-fibered stock, such as bleached sulfite or bleached sulfate. Often direct entry grades are suitable. The stock must be refined moderately in order to develop the necessary folding endurance, and reduce the amount of fuzz on the sheet, but jordaning of the liner stock is held to a minimum in order to preserve the bending qualities. In comparison, the filler stock, which is usually derived from mixed wastepapers, is customarily refined quite heavily in order to eliminate lumps of undefibered stock or fiber bundles, which may have passed the cleaning and screening system. The principal grades are bleached manila, white-patent coated, and on-machine clay-coated. They are used for packaging cereals, soap powders, cigarettes, wearing apparel, and similar items. Specifications may include weight, caliper, bursting strength, brightness (top liner), sizing, tensile, dirt count, moisture content, and color. Printing qualities are of great importance, and, because most of the board is used in packaging, the gluing properties are also very important.

Combination Manila Board—Another grade, known as combination manila board, designates a board wherein one or both of the outer plies are of a different raw material and/or color than the middle ply, whereas a plain or solid board has the same material throughout. The filler can be composed of approximately 40% unbleached sulfite and 60% mechanical pulp. Clay coating may be used. It is generally used for cartons with high-gloss printing.

Container Board—This board is made with a filler of mixed wastepaper and the liner made of kraft fiber, which may be all virgin kraft or a mixture of virgin kraft and waste kraft. If waste kraft is used, the board is sometimes referred to as jute liner board. Jute liner board may be given either a dry finish or a water finish, and in some cases the board is calender sized with starch, carboxymethyl cellulose, and so on. Filled kraft liner board is similar to jute liner board, except that virgin kraft stock is used in the liners. These grades are made in the standard thicknesses of 12, 16, or 30 points.

Setup Board—This board differs from folding-box board in that bending quality is not required. It is made of short-fibered stocks, such as groundwood, news, straw, or mixed papers. In some cases, the liners may be made from bleached white stock in order to improve the appearance. The board is used for rigid boxes and may be a solid or combination board, depending on the style of box; it ranges in thickness from 0.405 to 1.650 mm (0.016 to 0.065 in.) and weighs 290 to 1005 g/m² (60 to 150 lb/1000 ft²). Stiffness, rigidity, and resistance to abuse are essential qualities.

(continued)

SECONDARY FIBER PULPING 573

TABLE 4-69 Continued

Other Grades of Board—Other grades of cylinder board include laundry board, calendar board (usually a white-patent-coated board or a clay-coated board), cracker caddies (usually a jute-lined board), bottle-cap board (usually lined with sulfite stock), and matchbook board (either a bleached manila-lined board or a white-plate-coated board). In the case of milk-bottle-cap board, it is desirable to have a weak bond between two of the plies so that a tab can be readily raised on the cap when removing it from the bottle. Weakening of the bond can be achieved by adding wax emulsion to one of the plies at the point where the split is to occur. It is made of bleached prime pulp and normally has a thickness of 16 to 30 points, the actual caliper depending on the type and size of the container, which may be half point, quart, or half gallon.

Duplex Paper—Duplex paper is a special grade of two-ply paper that is made on either a two-cylinder machine or on a combination cylinder and four-cylinder machine. The product is used for big papers, for example duplex flour sacks that have a white ply on the outside and a dark blue ply on the inside. The blue ply is made from 100% rope and the outside ply from part rope stock and part sulfite pulp. Virgin kraft can be substituted for the rope if adequate refining is available. The inside ply is refined to a lower freeness than the stock for the outside ply and considerable trouble is often experienced in obtaining a good bond between the plies. It is necessary to have the plies very wet at their point of contact and to use graduated pressure on the wet presses in order to reduce the danger of crushing.

Backliners—On all the above grades of board it is possible to use a backliner of different stock between the filler and the top liner. This backliner helps improve the cleanliness, brightness, and formation of the board, and hence is effective in improving the printing surface. The object is to cover up the dark filler stock and produce a better foundation for the liner stock. News stock is a common grade for use as a backliner, and a board made in this manner is said to have a "skin news backing." Manila has also been used as a "skin."

Backliners—On all the above grades of board it is possible to use a backliner of different stock between the filler and the top liner. This backliner helps improve the cleanliness, brightness, and formation of the board, and hence is effective in improving the printing surface. The object is to cover up the dark filler stock and produce a better foundation for the liner stock. News stock is a common grade for use as a backliner, and a board made in this manner is said to have a "skin news backing." Manila has also been used as a "skin."

1. Black Clawson Lo-Intensity Pulping (LIP)
2. Voith-Morden Turbo Separator System
3. Beloit Jones Belcor System
4. Escher Wyss Fiberizer System.

They are described in the following pages.

Black Clawson Lo-Intensity Pulping System. This is a 1135,1136, relatively new system for secondary fiber processing. It requires minimum energy input, about 36 kWh (2 hpd) per ton, which makes for low operating cost. It results in minimum degradation of contaminants, thus making their removal more effective. The pulping system, which is shown in Figure 4-125, operates continuously. It depends on extraction through 1.59- to 2.54-cm (5/8- to 1-in.) holes and requires only 18kWh (1 hpd) per ton for defibering. The waste paper normally used in this pulper contains about 5% contaminants, so even if the system is 100% efficient, there will always be 5% contamination in the stock slurry. The ragger performs the function of continuous removal of strings and rags, and the junk box removes the heavy contaminants that are too large to pass the extraction plates holes.

The extracted stock is pumped through centrifugal cleaners, which remove the heavy contaminants. The pressure-drop across the cleaner should be 69 to 103 kPa (10 to 15 psi) and, because of the high volume of rejects, an automatic reject dumping system is provided. The stock then enters a pressurized power screen equipped with 0.2- to 0.3-cm (0.079- to 0.125-in.) holes powered by 22.5 Kw (12.5 hp) at 4% consistency. The defibered stock accepted by the screen moves downstream for additional cleaning and screening.

The reject flow from the pressure screen amounts to 40% of the fiber weight and contains a certain amount of defibered stock, which acts as a vehicle to carry the contaminants. The reject flow at about 5% consistency passes to a defibler, which is an adjustable clearance machine designed to handle the large un-defibered flakes of paper and contaminants. The defibler has no bar-to-bar contact so hydraulic shear alone acts on the paper flakes but leaves the contaminants intact. The defibler uses 18 to 27 kWh (1 to 1.5 hpd) per ton. The reject flow continues to a vibrating screen, equipped with 0.47- to 0.95-cm (3/16 to 3/8 in.) holes at 3% consistency. The accepts from the screen are turned to the pulper and the light rejects are rejected for disposal.

Voith-Morden Waste-paper-pulping System. This system consists of a pulper, turbo separator, high-pressure cleaner, and vibrating screen.¹¹⁷ It is designed for continuous operation and it removes both heavy and light contaminants with minimal loss of usable fibers. It washes and cleans the fiber, so that little or no additional screening or cleaning is needed downstream. It operates at 3 to 4% consistency and requires about 36 kWh (2 hpd) per ton.

As shown in Figure 4-126, the pulper receives the baled waste paper from the

SECONDARY FIBER FULPING 575

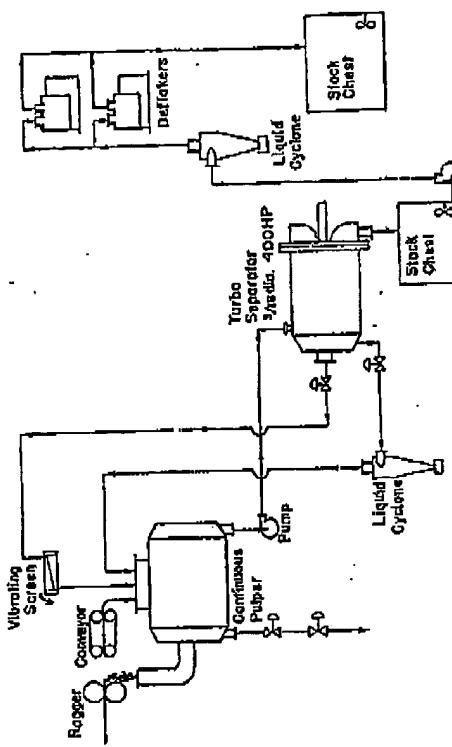


Figure 4-126. Turbo-separator system by Voith-Morden Co.

conveyor. Lightweight contaminants collect in the turbulence zone, form a rag rope, and are removed by a time-controlled ragger. Heavy contaminants collect in the junk trap. Partially defibred secondary fiber plus floating contaminants are pumped into the extraction plate. They are pumped into the turbo separator for further defibering and cleaning. A large cup on the rotor stabilizes the vortex, entraining the remaining lightweight contaminants. Accepts pass through the rotor-extraction plate, with heavy contaminants being removed continuously (tangentially). A cleaner receives the light contaminants and return the accepts to the pulper. Lightweight contaminants are periodically discharged and pumped to the vibrating screen, which returns accepts to the pulper. Downstream deflaking and screening completes the system.

Beloit-Jones Belcor System. The Belcor System, as shown in Figure 4-127-118, is a combination cleaning and defibering system arranged for continuous extraction. The stock from the pulper is pumped into a Belcor unit continuously and tangentially. Its function is to screen out intermediate-size pieces of plastic and wet-strength paper; to serve as a secondary pumping unit after virtually all large pieces of plastic and heavy junk have been removed; and to remove any heavy metal, such as paper clips and staples, that have not been separated in either the pulper or cleaner. Referring to Figure 4-127, the light rejects are discharged at the central outlet (2) opposite the rotor. Usually a flow rate of 10% by volume is adequate to prevent an excessive buildup of rejects in the tank and to insure good cleaning efficiency. The reject flow may be controlled by a hand-valve (3) or inlet pressure. The flow is adjustable to the amount of rejects in the waste. The rejects are screened on a vibrating screen (4) and returned to the

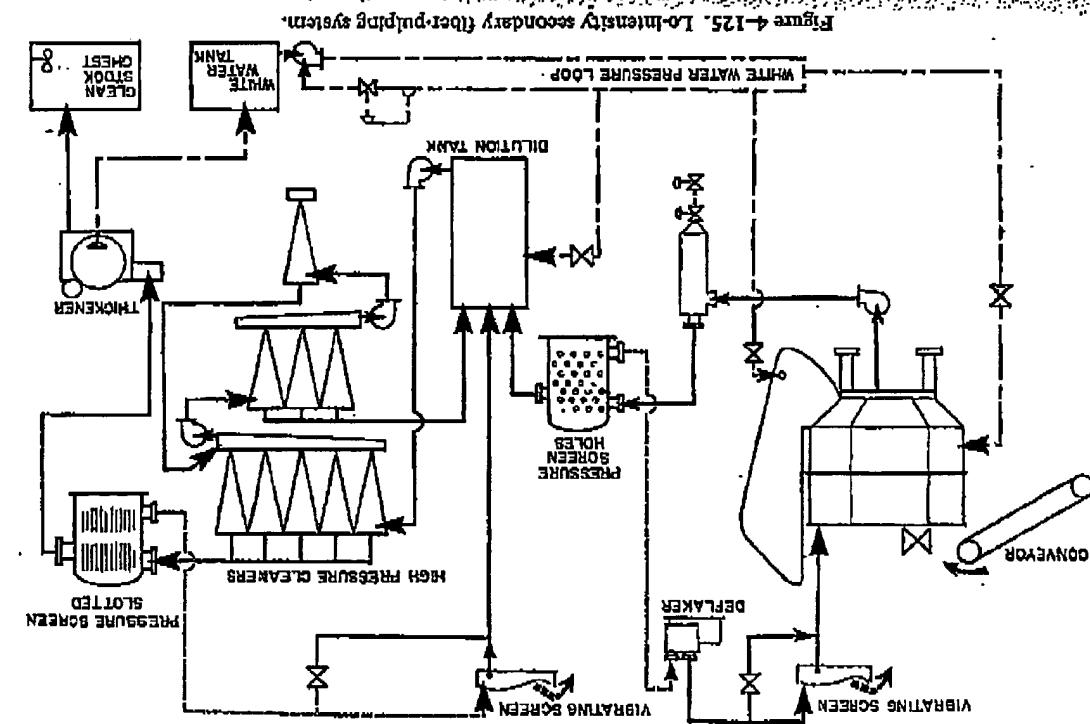


Figure 4-127. Low-intensity secondary fiber-pulping system.

SECONDARY FIBER PULPING 577

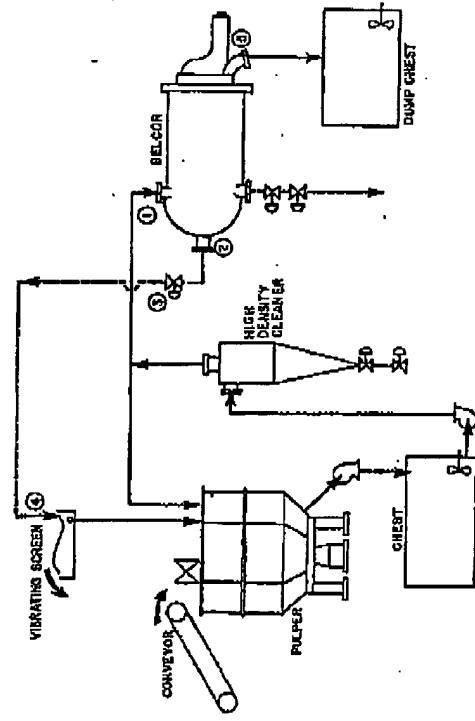


Figure 4-127. Beloit-Jones Belcor system.

pulper. The accepted stock is discharged through a perforated extraction plate behind the rotor (5). Hole sizes range from 0.317- to 0.472-cm (1/8 to 3/16 in.) at stock consistencies of 2 to 3 %.

The Belcor unit serves as a second pulping stage. Its defibering capacity is approximately the same as a conventional pulper and it uses 9 to 18 kWh (0.5 to 1.0 hp) per ton. The primary pulper, which has large diameter holes, requires low power because some of the pulping power is applied at the Belcor. The cleaning efficiency depends on particle size and concentration of the contaminants and ranges from 75 to 90%.

The Escher Wyss Fiberizer System. This system, which is shown in Figure 4-128, 1139 was designed to separate out contaminants present in wastewater stock as it leaves the pulper. Within the Fiberizer the functions of centrifugal separation, screening, and deflaking are combined. Stock enters the fiberizer through a tangential inlet in the conical housing (1). Rotational movement of the stock is caused by the rotor, which is adjusted close to the screen plate. Fixed deflaking bars are positioned in a circle around the rotor to improve the deflaking action and to keep the screen clean by turbulence. Accepted stock leaves through the screen plate, which has 0.3- to 0.4-cm holes (2). Large, undeflaked flakes are retained along with the plastic film and heavy contaminants. Stock containing a high percentage of light contaminants is drawn off intermittently through an outlet in the center of the cover (3). Heavy contaminants are collected by centrifugal force in the junk trap at the bottom of the unit (4) and are dumped automatically at selected intervals. A percolating flow of water washes back any fibers. The adjustable gap between

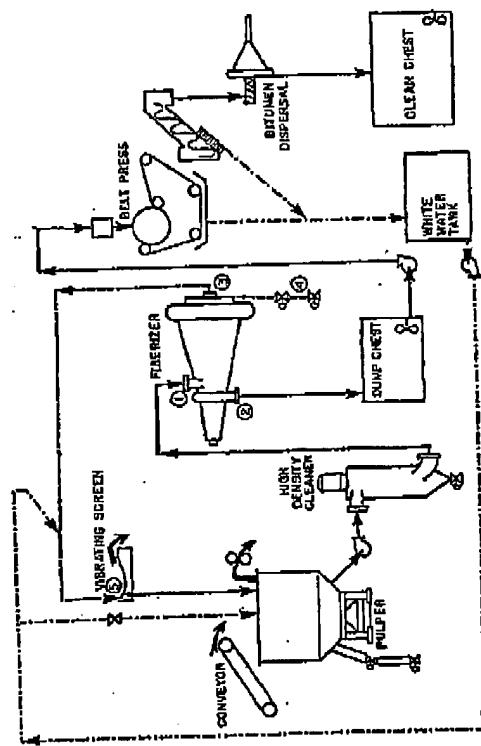


Figure 4-128. Escher Wyss Fiberizer system.

the rotor and fixed plate as well as the deflaking bars defiber the large flakes, but the cutting of thin plastic foil is prevented. The lightweight contaminants, such as foil and polystyrene, are expelled intermittently through a control valve to a vibrating screen (5).

Bituminous Asphalt Dispersion. Bituminous asphalt, which is commonly used as a laminating adhesive and as a water-vapor barrier in sack papers, fiber barrels, containers, and kraft shipping bags has a long history for causing problems in recycling. When these papers were used in older systems, they produced black-pulped. When these papers were created. Considerable effort has gone into the development of improved systems. 1140-1142 It is possible to disperse the asphalt to adjoining fibers during the secondary fiber pulping so it is not noticeable in the finished product and will not produce bleeding in papers, such as dry-wall gypsum board. The most commonly used system in the United States is shown in Figure 4-129. After the thickener in a continuous wastewater system, such as that illustrated in Figure 4-124, the stock is dewatered to 12 to 16% with an inclined-screw thickener, pressed to 35% consistency in a cone-type press, and discharged by means of a feed conveyor to a continuous digester. A plug of stock at about 50% consistency is formed in the throat of the feeder, which helps to contain the steam pressure in the digester tube. Steam at about 515 kPa (75 psi) is introduced into the continuous digester, pressed to 35% consistency, to a continuous digester. As the temperature of the stock well above the melting point of the asphalt. As the temperature of the stock well above the melting point of the asphalt. As the stock is turned by the action of the internal flights of the digester tube, the softened asphalt is deposited on and spread over the surface of the fibers. The digester tube has a variable speed drive that controls the exposure time of

3. Washing.
4. Dewatering or thickening.

Ink removal is sometimes accomplished through flotation. Bleaching and bleaching agents are included if required to produce white pulp.

Types of Papers Used in Drinking

The quality of deinked pulp is primarily determined by the kind of waste paper used in the deinking plant. Thus it is desirable to obtain only the brightest grades of wastepaper, all of the same general kind. In some cases, wastepaper can be obtained from large paper-using plants that is so well segregated that it requires no sorting. The trend is away from hand-sorting (because of excessive labor costs) and toward the use of deinking methods based on the proper balance of chemical and mechanical operations to produce the desired end results from a variety of papers. The most desirable papers for deinking are fine shavings, cuttings, ledger stock, and magazine stock. The lower grades of mixed papers can be sorted to remove undesirable papers and other contaminants, such as carbon paper, waxed papers, impregnated papers, glassine, parchment, bits of cloth, typewriter ribbon, bits of wood and dirt, highly colored covers and posters, and wetstrength papers. But sorting is costly and disappearing from the industry.

Purchase specifications for wastepaper for drinking sometimes limit the groundwood content because groundwood is difficult to drink; it turns brown during the drinking, and it cannot be bleached with a straight hypochlorite bleach. Old groundwood papers are particularly difficult to desifer and tend to

form small, hard clumps of fibers. Originally, groundwood papers were excluded by most drinking mills, but with the more widespread utilization of groundwood in printing papers and other grades, it has become increasingly difficult to obtain enough groundwood-free wastepaper to satisfy the demands of drinking plants. Therefore, special processes have been developed to handle groundwood papers. When the groundwood content exceeds 10%, the deinking method must be designed to handle groundwood. Under these circumstances, a special bleaching process is required to bring brightness levels to 70 or more. Papers containing groundwood can be identified by the color reaction obtained when the paper is stained with aniline sulphate (yellow), phloroglucinol (red), or a solution of sodium hydroxide (yellow). West-strength paper is objectionable because of the resistance of the paper to fiber separation, and high temperatures and low pH are required for the disintegration of these papers. Glassine and parchment papers are difficult, if not impossible, to defilter. Waxed, resin-impregnated, and resin-coated papers are resistant to water and cannot be defiltered by ordinary deinking methods. Cellophane will not disperse, but unless it is excessively

Papers treated with rubber-like or thermoplastic materials cause problems in drinking. As little as 1 kg of rubber-like material can ruin over 100 ton of brittle, it will remain large enough to be removed by screening.

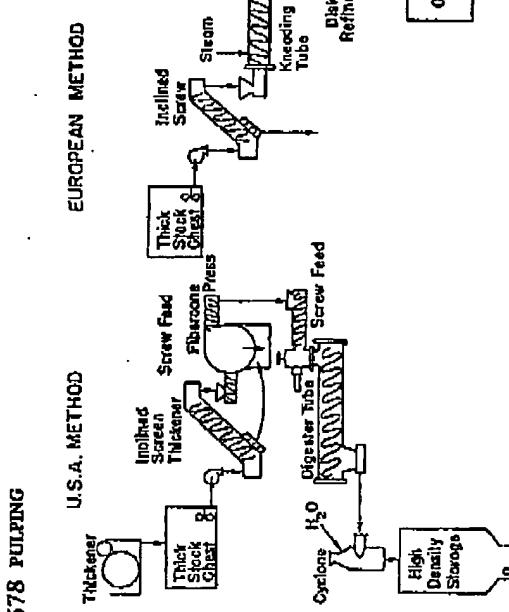


Figure 4-129. Bitumen asphalt dispersal systems, showing methods used in the United States and in Europe.

the stock to the steam and the tumbling action. At the discharge end of the digester tube, a special orifice releases the stock from the pressure vessel to atmospheric pressure. This accompanying steam propels the stock to the cyclone, where dilution and cooling water is added to return the stock to the desired temperature and consistency. The power required in this system is 59 kWh (3.3 hp) per ton; the steam requirements are 0.4 kg (0.9 lb) of steam/0.45 kg (1 lb) of fiber.

In the European method a different approach is used as shown in Figure 4-129. The stock from the thickener is dewatered and added to a kneading tube, where in the presence of steam, the stock is thoroughly kneaded to disperse the asphalt. A disk refiner at the discharge end of the kneading machine disperses the asphalt thoroughly among all of the fibers.

Deinkine of Old Planets

In order to produce a white pulp from wastepaper that will be suitable for book and other similar papers, it is necessary to remove the ink from the wastepaper. The process of doing this is known as deinking. There are two basic steps in deinking: (1) dissolving or loosening the ink by chemical means and (2) removing the ink from the pulp by mechanical washing. All deinking systems have the following stages:¹⁴³⁻¹⁴⁵

1. Pulping or desilbering in the presence of chemicals.
2. Cleaning and screening.

pulp^{119,120} if it is not properly dispersed. Pigment-coated book papers are readily delinked. Colored papers present somewhat of a problem in delinking, particularly if the dyes used in the paper are resistant, or fast, to chemicals. Most of the basic and acid dyes are destroyed by a caustic cook and can also be reduced with zinc hydrosulfite, although basic dyes tend to reoxidize on long standing. Most of the direct dyes can also be stripped with either caustic, chlorine, or hydrosulfite, but there are exceptions, such as the stilbene yellows, oranges, and turquoise blues, which are regarded as nondelinkable.¹²¹ The pigment types differ: (1) the chrome yellows and iron blues are destroyed by caustic, (2) the azoic types are regarded as nondelinkable, but can be discharged by direct chlorination, and (3) the phospho-tungstic-molybdc lakes present no problem since they are easily destroyed by hypochlorite bleach.

Chemicals Used in Delinking

Most delinking is done with plain alkali, but detergents and dispersing agents, such as soaps, silonated oils, bentonite, sodium metasilicate or silicate pentylate, and other surface-active substances are sometimes used in combination with alkali. An ideal delinking formula would include an alkali to saponify the varnish or vehicle of the printing ink, a detergent to aid in the wetting of the pigment in the ink, a dispersing agent to prevent agglomeration of the pigment particles after release from the paper, and an absorption agent to bind the pigment and prevent redeposition on the fiber.

Alkali is used in the delinking formula for two purposes: (1) to remove resin staining from the paper and (2) to saponify the ink vehicle and release the pigment in the ink. There is generally about 0.5 to 2.0% alkali on the weight of the paper; this must be completely removed if white pulp is to be produced. From the standpoint of ease of delinking, there are four principal types of inks:

1. Drying, oil-base inks.
2. Non-drying, oil-base inks.
3. Inks having a synthetic resin base.
4. Metallic inks with latex base.

Drying, oil-base inks that are slightly oxidized can be readily saponified by alkali. However, completely oxidized oil-base inks; nondrying, oil-base inks; and inks having a synthetic resin base cannot be completely saponified by alkali of ordinary concentration. Consequently special methods of delinking must be used for papers containing these inks. The various high-gloss and metallic inks in use today are extremely difficult to remove with alkali. Solvents (e.g., tr- or tetra-chloroethylene, benzene, or carbon tetrachloride) or soaps and detergents can be used to aid in the delinking of these papers. Rosin is readily removed by saponification with alkali, and even the waxes used in sizing paper are melted and readily removed. Solvents are sometimes used to remove wax and polyethylene.¹²²

The type and amount of alkali required in delinking depends on the type of mechanical treatment and the temperature and time of cooking. Sodium carbonate and sodium hydroxide are widely used in delinking. Sodium carbonate is a milder agent than sodium hydroxide and results in less oxidation of the fiber and less fiber loss. Sodium hydroxide results in faster delinking. However, too strong caustic soda may seriously attack the cellulose, since it is in a particularly susceptible condition, having a high area of surface exposure. About 5% sodium hydroxide is the maximum used. A typical caustic soda delinking formula for white ledger paper is 3% sodium hydroxide solution at a temperature of 71° C.¹¹² Concentrated sodium hydroxide should always be diluted if it is added directly to the fibers. A concentration greater than 17% should never be added to a mixture of fibers and water because caustic of this concentration will dissolve the cellulose and contaminate the batch with sticky scabs, similar to soft, hot melts and tattes. If strong caustic must be used, it should be added to the pulper batch water before the paper is added. If used alone, 3 to 8% sodium carbonate is sufficient for the delinking of most papers. Some prefer a mixture of 2.5% sodium carbonate and 0.5% sodium hydroxide.¹¹² Only a small part of the alkali is consumed in the cook. Laboratory results have shown that the consumption of alkali is in the range of 0.25 to 1.0%, based on the weight of the paper,¹¹⁵ so that most of the original alkali appears in the waste liquor. In delinking ordinary rosin-sized papers, the alkali reacts with the rosin to form a rosin soap, which acts as a detergent for the ink particles. Sequestering agents may be helpful in preventing the formation of calcium soaps. If unsized papers are used, no rosin soap is formed, and special detergents may be helpful. Plain soap appears to be helpful under some circumstances.¹¹⁶

Sodium silicate is sometimes used for part of the alkali. Silicate is effective at a lower pH than sodium hydroxide. This is important in the delinking of groundwood papers to prevent yellowing. The more alkaline grades of silicate are preferred. A suitable grade is one containing 1 part Na₂O to 1.6 to 1.7 parts SiO₂, or sodium metasilicate (Na₂ SiO₃ · 5H₂O). The amount of silicate varies from 2% to 9%. High-grade wastepaper can be delinked by using 3% of 42° Baume (Bé) silicate and 1.5% sodium hydroxide at a temperature of 66°C for 45 min.¹¹⁷

Once the ink pigment has been released, dispersing and absorptive agents are desirable to prevent the pigment from becoming redeposited on the surface of the fibers, which are strongly absorptive. One patented process calls for a mixture of high-silica sodium silicate and a fatty acid.¹¹⁸ Bentonite may be used for the same purpose¹¹⁹ and to prevent the agglomeration of carbon-black particles around curds of calcium or magnesium soaps formed during delinking. Other clays may be used, or the clay may be obtained from the filler in the paper. Waste book and magazine stock have been successfully delinked for years, and part of the ease of delinking these papers can be attributed to the presence of clay fillers. Bragg¹²⁰ points out that 0.75% bentonite completely absorbs the ink and scum in a pulp containing unsaponified ink. Removal of ink by absorption

tion on a hydrophobic particulate solid has been shown to be effective,¹¹⁶¹ but any soap or other surface-active agent must first be deactivated.

Peroxide has been proposed as an aid to delinking operations. Peroxides are particularly effective in combination with silicate. A suitable formula is 1 to 3% hydrogen peroxide, 3 to 6% silicate (58.5°Bé, 1.6 ratio), and 0.5 to 2.0% sodium hydroxide to be used at a temperature of 71°C for a period of 35 to 90 min.^{1162,1163} If sodium peroxide is used in place of hydrogen peroxide, no sodium hydroxide need be added. Regular alkaline liquors have a detrimental effect on the brightness of groundwood containing pulps, but peroxide helps to prevent darkening and color reversion of the pulp. There is no advantage in using peroxide, unless the groundwood content of the waste paper is over 15% or thereabout. Zinc hydroulfite has also been used in delinking groundwood papers to improve the brightness. It is used to aid in the destruction of dyes when colored papers are present. About 0.5 to 1.5% will improve the brightness of groundwood papers.

Most of the formulas used in commercial delinking processes are relatively simple. For colored ledgers, tab card, computer printout, and other selected chemical fiber waste, some typical formulas are:

1. Sodium hydroxide, 4%.
2. Sodium hydroxide, 2.5%, plus 2.5% sodium silicate, plus 3% sodium carbonate.
3. Sodium hydroxide, 3%, plus 2% sodium silicate.
4. Sodium hypochlorite, 0.8%, expressed as chlorine, plus 4% sodium hydroxide.

For colored ledgers and for coated carbonless papers that are made with activated clay and encapsulated ink, formula number 4 is best. The hypochlorite is used to strip the color. It is added to 60°C water and the batch delinked for 5 min. The sodium hydroxide diluted to not more than 15% concentration is then added and the delinking continued for an additional 15 min. Tab card and ledgers of low groundwood content can be processed in formulas 2 or 3. Newspapers can be delinked in solutions containing (1) 2% sodium peroxide, plus 5% sodium silicate, (2) 2% sodium peroxide, plus 3% sodium silicate, plus diatomaceous earth, or (3) 1% sodium hydroxide, plus 1.5% sodium silicate, and 0.7% hydrogen peroxide. The temperature should be kept below 54°C when delinking groundwood. A formula for the delinking of groundwood papers that can be used at elevated temperatures is 1% sodium hydroxide, plus 8% sodium perborate, plus 2% sodium carbonate. After pulping, the stock is dewatered to 20% consistency with inclined-screw extractors, and rediluted for cleaning in high-pressure-drop centrifugal cleaners and screened through pressure screens having 0.014-inch slots. Care must be taken not to devitalize the stock beyond 20% density because the ink, which is partially saponified and soft, will blacken the fibers by the rubbing pressure.

Pulping Conditions Used in Delinking

Pulping may be either batch or continuous. The batch method permits better control of water, chemicals, and waste paper added to the pulper. The batch, at a predetermined consistency, temperature, and chemical concentration is processed for a predetermined length of time, thus providing positive control of pulping conditions, regardless of the size of the perforations in the pulper through which the stock is to be withdrawn. Delinking is generally 98 to 100% completed and sufficient time is allowed for chemical reaction.¹¹⁶⁴⁻¹¹⁶⁶ Samples can be taken before dumping the batch to insure that the pulping is complete. Overall, the batch process is considered the best for delinking, even though productivity will be 25% less than that obtainable with continuous operation.

Continuous pulping allows maximum productivity for a given size of pulping unit. Water, chemicals, and waste paper are added at a controlled rate commensurate with the pulping capacity and the system demand. The waste paper is exposed to the chemical solution and to the delinking action of the rotor until the particles are small enough to be withdrawn through the holes in the extraction plate. Retention time in the pulping unit varies and not all the waste paper has the same exposure to the chemical action and delinking action.

The degree of delinking is dependent on the rate of extraction and the demand. Sampling is possible but is not always representative of the entire batch.

Originally delinking was done by cooking in globe digesters at consistencies ranging from a low of 6% to a high of 35%, temperatures of 79 to 93°C and cooking time of 3 to 8 hr. Steam was generally introduced directly into the charge by means of a perforated pipe running throughout the width of the boiler. Heating with saturated steam rather than superheated steam was used, particularly if sodium hydroxide was used as the cooking agent.¹¹⁶⁷ In extreme cases as long as 8 to 10 hr at 275 to 345 kPa (40 to 50 psi) steam pressure was used,¹¹⁶⁸ but this resulted in a loss of yield due to degradation and solution of the carbohydrates. At the end of the cooking period, the stock was dumped, in the form of a pulpy mass, to a pit underneath the cooker, where the liquor was drained away. In most cases the liquor was saved for reuse. Most delinking is done in open pulpers at temperatures of 60 to 82°C and a retention time of 45 min to 1 hr 30 min. The power required is used for delinking; there is no actual "pearing" of the stock in the sense of "hydrating" the fiber. Pulping consistencies vary from 7 to 25%. High-consistency pulping speeds up delinking, reduces the amount of chemical required, and results in pulp of higher fineness, compared with low-consistency pulping. Cooking liquor can be recovered by extraction immediately following the dump chest. A typical flow diagram is shown in Figure 4-130 and a shortcut system is shown in Figure 4-131.

The use of a rotating drum that is perforated along part of its length has been proposed for delinking and prescreening of waste paper, generally using 1 to 2% sodium hydroxide.¹¹⁶⁹

After cooking, the quality of the delinked stock can be evaluated by making hand sheets and examining by both reflected and transmitted light. Particles

SECONDARY FIBER PULPING 585

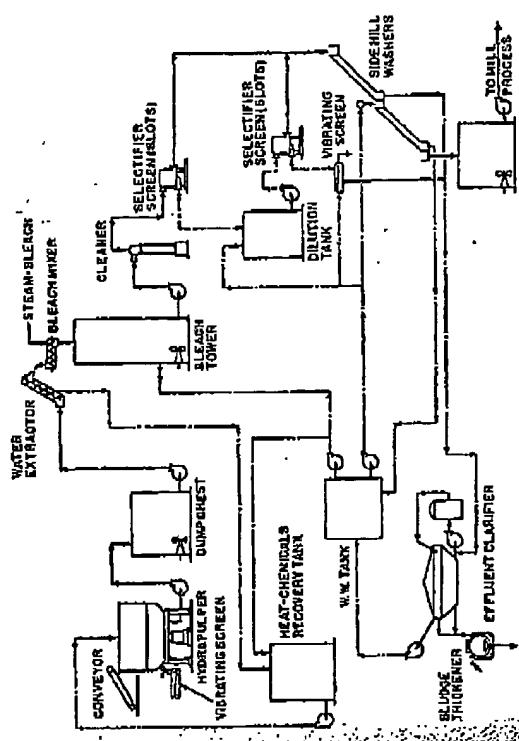


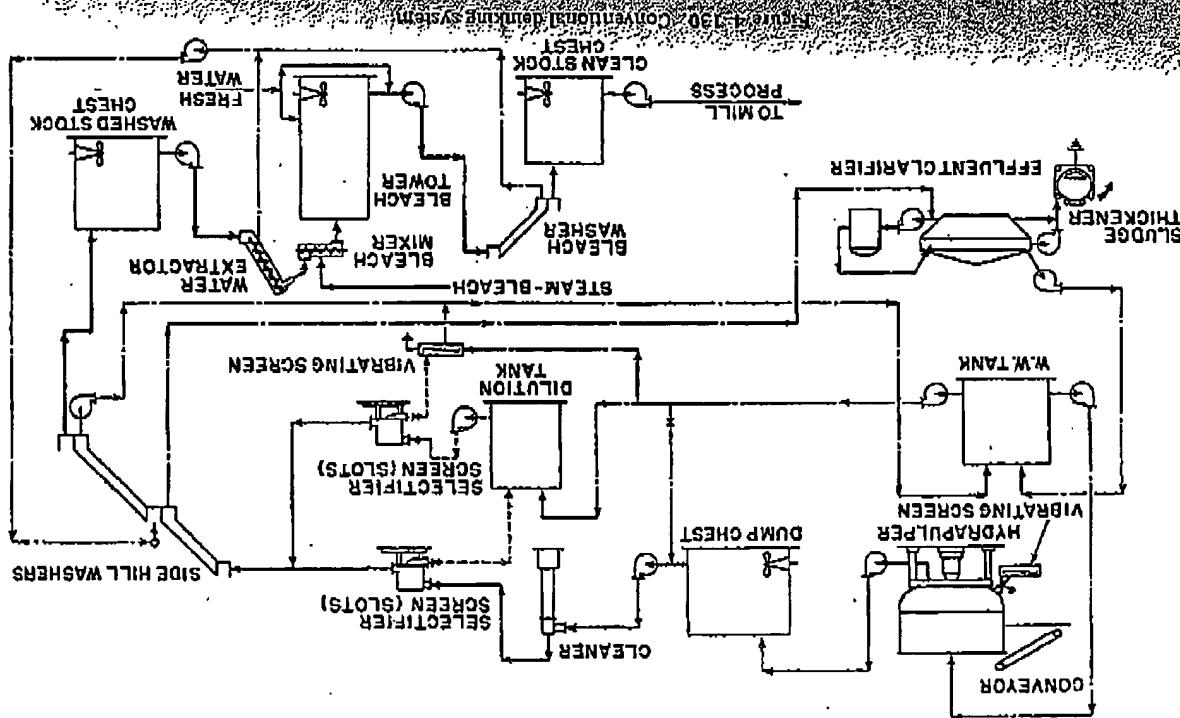
Figure 4-131. Shortcut deinking system.

In the sheet should be examined to determine their source. Some of the most undesirable materials are ink balls (ink from papers used by the printers for platen), plastic particles (from adhesives and coatings), latex or oxidized rubber particles (from impregnants or binders), asphalt (from laminated papers), dispersed paper particles (from high groundwood content or wet-strength papers), and hot melt adhesives (from the bindings of cups and square-edge magazines).

Cleaning Stage of Deinking Process

The cleaning step follows the defibering of the wastepaper in the pulper. Centrifugal cleaners, which act on the principle of converting pressure to velocity, thus providing the centrifugal force for separation, are the type generally used. The stock enters the top of the cleaner. While gravitational force remains constant, the velocity of the stock is slowed down because of friction with the sidewalls. The clean stock returns up through the center area of low or no pressure and is discharged at the top of the separator. The heavy contaminants separate at the bottom of the cleaner and are removed.

The two important variables in conical separators are stock consistency and pressure-drop in the separators. High-pressure-drop cleaners, having a pressure differential of 205 to 275 kPa (30 to 40 psi), which are used at consistencies ranging from 0.4 to 0.8%, will remove metal matter, sand, large particles of ink,



SECONDARY FIBER PULPING 587

and small contaminants. They are used in multistage arrangement after low-pressure-drop cleaners in the process. Low-pressure-drop cleaners are used at a pressure drop of 48 to 138 kPa (7 to 20 psi) and a consistency of 0.8 to 5.0%. They are usually in single stage arrangement followed by screening and then high-pressure drop cleaners. Low-pressure-drop cleaners remove contaminants that are 0.42 cm (1/16 in.) in diameter and larger.

Later in the process, core bleed cleaners, such as Calleco and Tri-Clear, can be used to remove materials of very low specific gravity, such as plastic particles, hot melts, and metallic inks. These operate at 0.7% consistency and below.

Screening Stage of Dewatering Process

After centrifugal cleaning, the stock is screened before washing. Pressure screens having small holes or slots are used. When maximum cleaning is desired, pressure screens in series are used, the slots or holes being smaller in the secondary than in the primary screen.

Primary screening can be done at consistencies ranging from 0.3 to 4.5% depending on the size of the hole or slot. Most primary screening is done to 1.0 to 1.5% consistencies. There are many types of pressure screens. Most operate at inlet pressures of 138 to 275 kPa (20 to 40 psi) and a slot size of 0.036 cm (0.014 in.). The reject flow from the primary screens should be diluted and rescreened to increase the yield. Fine screening is done by means of rotary vibrator screens that vibrate at high frequency, while the cylinder is partially submerged in the stock. These screens have slots ranging from 0.010 to 0.020 cm (0.004 to 0.008 in.) in width. The consistency is generally 0.8 to 1.0%. Rejects are disposed of as refuse or passed to a tailing screen. The reject flow from both the primary and the secondary screens are combined and passed to the washers.

Washing Stage of Dewatering Process

The stock after cleaning and screening must be washed to remove the dispersed inks, clay, and chemicals. The type of washing depends on individual preference, water availability, effluent-handling system, and initial investment limitation. There are five different methods of washing. These are:

The Lancaster Washer.

Sidehill Washer.

Inclined-Screw Washer.

Vacuum Washer.

The American Disk Filter.

dispersed the ink is in the stock. Typical washers remove about 55% of the ink from the stock. Theoretically, it is possible by repeated washings to remove up to 99% of the ink, but in commercial practice this is not feasible. The ink particles are generally fairly large and tend to become entrapped by the fibers during the washing stage. Another effect is the redeposition of the ink particles on the fiber surfaces; this can occur in any washing operation. If dewatering and washing have been done well, stock from old magazines should contain not more than 3% inorganic solids, be completely free of large dark-colored material, and have a brightness of around 50 GE.119

The problems of waste water treatment from the washing stage in the dewatering process have deferred many aspiring companies from using a washing process, despite its usefulness in removing fillers and unwanted fines from the stock. Removal of fines is important to the subsequent papermaking process because below a certain size, fines cease to contribute to web strength; they reduce drainage on the paper machine; they increase the drying cost; and they cause dust and lint during printing of the paper. To treat waste water from the dewatering process satisfactorily, some means of treating the inky water is required, preferably by means of ink-sludge concentration. Large quantities of water are required to wash the pulp thoroughly. Excessive washing has the disadvantage of causing large fiber losses, but the pulp that is insufficiently washed will darken when alum is added because of the precipitation of suspended impurities on the fibers. Foaming should be held to a minimum, since the foam and froth formed during washing has a strong affinity for carbon-black particles. If the foam is allowed to break on the mat of stock, it may deposit particles of carbon, thereby resulting in a darkening of the stock. Antifoams are sometimes added to keep down foam.

Lancaster Washer. In this method a revolving cylinder covered with wire mesh (20/20, 40/40, 40/60 depending on wastepaper) is partially immersed in the dilute stock, and a mat is formed on the cylinder surface due to the controlled differential head. The water, ink, and clay flow from the outside into the center and discharge through the ends of the cylinder. The differential head is controlled by adjustable weirs in the discharge boxes. It is considered the best method of washing because the mat is thin and allows the water to flow freely into the center of the cylinder. A couch roll is used to aid in water removal; this pressed-out water combines with the naturally drained water. The pressing action dewateres the mat to 8 to 10% consistency. The stock is transferred from the cylinder face to the rubber-covered couch roll and a metal or mica-clipped doctor removes the mat from the couch roll.

Lancaster washers are costly and space-consuming. Their capacity is low, about 0.020 to 0.034 m^3/min^2 (5 to 9 gal/ft^2) of cylinder area can be realized. Drainage rate is controlled by stock temperature, wire mesh, fineness of the stock, and differential head. A 594-cm (60-in.) diameter Lancaster will handle 0.14 ton/cm (0.36 ton/in.) of face; thus a 152 by 594-cm (60 by 234-in.) face will handle 85 ton of pulp. Two or more Lancasters arranged in series

SECONDARY FIBER PULPING 589

with repulper mixing chambers between them can be used for multiple washings. Losses are in the range of 8% for the first stage and 6% for the second stage.

Sidehill Washer. The sidehill, or slide-wire washer, considered by many as a crude type, does a very good job of washing. The stock at a consistency of 0.6 to 1.0% enters a headbox at the top of the washer, the velocity is damped by a weir, and the stock then overflows onto the inclined, wire-mesh surface, which is inclined at an angle of 38°. The water drains through the wire, and the stock slides or tumbles down the inclined-wire surface. The water is collected in a compartment under the wire, which extends to the bottom of the washer. As the fibers tumble down the inclined wire, new areas are constantly exposed for water removal. The wire mesh is usually 58 X 80 or 58 X 100 and in some rate instances 60 X 60. The stock is collected at 3 to 7% consistency in a discharge box at the bottom of the washer.

The sidehill washer is characterized by low initial cost, low operational cost, and low maintenance cost. Freezes of the stock has little effect on the operation, compared to mat-type washers. Sidehill washers are commonly made in a length of 3.65 m (12 ft) and a width of 9.14 m (30 ft). Such a washer will handle 100 ton of stock, or 0.1 ton/cm (0.28 ton/in) of face. Sidehill washer losses can be 10% in the first stage and 4% in the second stage, but this naturally depends on the type of stock being washed.

Inclined-Screw Washer. The inclined-screw washer consists of an inlet section that supplies stock to several inclined screws housed in a common casing or perforated cylinder having holes of 0.157 cm (0.062 in.) in diameter. As the screw rotates, the stock is pushed upward, the water is drained through the perforated cylinder, and the thickened stock is pushed out a separate discharge port, which is opposite the inlet section. The screw may have nylon brush on the leading edges to keep the perforated holes clear or, in some models, this is achieved by maintaining a very close mechanical clearance. The screw carries the ever-thickening stock upward to a point where the flight screw From this point to the discharge opening only the pressure of the upcoming stock pushes the thickened-stock plug toward the discharge opening. A thick plug of stock leaves the perforated cylinder, a special breaker arm, which is secured to the shaft of the screw, breaks up the plug and causes it to fall into the discharge opening.

Inclined-screw thickeners come in 22.8-cm (9-in.) and 40.6-cm (16-in.) diameters. The capacity of a 22.8-cm (9-in.) size is nominally 20 ton and the outgoing consistency ranges from 16 to 25%. This method of washing does not approach the Lancaster or Sidehill efficiency because ink particles are trapped in the 7.6-cm (3-in.) thick mat of stock and cannot be expelled with the effluent.

Vacuum Washer. Vacuum washers are used for continuous operation.

high productive capacity. A drum is immersed in the stock slurry and a vacuum applied to the submerged portion, causing the fibers to deposit on the outer surface as the drum rotates. The drum is divided into compartments that are connected to a rotating valve. Vacuum is applied through the valve to remove the effluent of ink, clay, and water. Several compartments may be connected to the vacuum port to form a progressively thicker and thicker mat. As the compartment leaves the vacuum area, air is permitted to enter to assist in the stock removal.

Water sprays may be used on the drum surface to assist in washing. Discharge consistency can be 12 to 17%. The drum washer does a reasonably good job, but due to the thick mat of fibers, some of the ink and clay particles are entrapped. Fiber loss is very low.

Disk-Type Washer. The disk washer operates on much the same principle as the drum vacuum cylinder. The washing areas are arranged in disks rather than a large drum. Each disk may be independently removed without disturbing the other disks and each disk can be operated as a separate washer. The disk has eight or more segments that are under vacuum as they pass through the slurry, thus forming a heavy mat on each side of the washer surface. As the segments pass the last vacuum port, air is introduced into the center of the segment and jets of water are applied to loosen the stock mat and roll it from the disk surface. The jet also cleans the wire or cloth surface.

The disk washer has a high dewatering capacity, which ranges from 0.028 to 0.020 l/cm² (2 to 5.0 gal/sq ft) of area. It has the same disadvantage as the drum vacuum washer, in that the heavy mat acts as a filter to retain ink and clay particles.

FLOTATION DENSITING AND WASHING SYSTEM

The flotation washing system consists of a cell or tank, a high-speed agitator, an overflow for froth removal, a mechanical paddle for removing the froth, and a discharge pipe to pass the stock to the next cell in the flotation line. The high-speed agitator induces a partial vacuum, which, in turn, causes air to enter the system and combine with the stock and flotation agents to form small air bubbles. Chemicals are used to create a suitable environment for the attraction of ink particles and pigments to the air bubbles, which are generated at the bottom of the cell and pass upward through the stock slurry to become froth at the surface. Because froth flotation depends on a difference in surface characteristics rather than density of the materials to be separated, it can be used for many materials regardless of their densities. As the ink-laden air bubbles reach the surface they are swept into a separate chamber by means of a two-armed rotating paddle that insures the removal of a uniform volume of froth from each revolution. The froth is withdrawn from the primary cells and pumped to secondary cells, where the ink is further concentrated. The fibers pass through the secondary units and are returned to the primary cells.

590 PULPING

The froth removed by the secondary cells is centrifuged to a secondary mass and disposed of. The water from the centrifuge goes to the effluent system. Although ink is more easily floated than fibers, it is necessary to pass the stock through a number of cells in series to remove all the ink. The foam from the primary cells must be passed through a number of secondary cells to reclaim the fiber.

Flootation is considered by some as the best system for recovering newspapers because all the fibers and fillers pass through the cell and only the ink is removed. The yield is higher than that obtained by washing, but there are disadvantages. The retention time required in a flotation cell is in the range of 13 to 15 min. The consistency is usually 0.8%, which means that thickening is required after flotation. Cuesta¹⁷ⁿ has pointed out that flotation and washing are both required for best results. In the case of fine paper, flotation helps to improve brightness, but washing is essential for pulp quality and clay removal. Flotation removes the species that are too small to remove by screening and too large to be removed by washing. In the case of news, washing is essential to obtain high strength and to obtain an increase in freeness. Flotation-washing is most efficient when applied to news or high groundwood papers but can also be applied to ledger, chemical pulp papers with slightly less efficiency. A complete system for pulping and recovery, using flotation, is presented below. The chemicals used for newsprint or high groundwood paper are 2% sodium peroxide, 4 to 5% sodium silicate, 0.1 to 0.3% foamer (Triton BASF), 0.05 to 0.25% foamer (Decolot R), and 0.3 to 0.8% Decolot S, or Biancol Colletor, all at a pH of 9.5. The chemicals used for chemical papers are 5% sodium peroxide and 0.8% sodium hydroxide at the pulping stage, plus the addition at the flotation cells of 3 to 5% fatty acid soap and 1% calcium chloride. Hot water and the chemicals for dispersing, flotation, and bleaching are added at the pulper before the wastepaper is added. This sequence is important because if the wastepaper is pulped first, before the chemicals are added, some printing inks could be beaten into the fibers, where they would not be floatable and thus impossible to remove. The formula needs to be exact because a definite alkalinity should remain for the following stages of the process. The pulped stock is dumped into a two-batch capacity chest, cleaned at high density and predeinked. This tends to separate foil, magazine glued with latex, wet-strength papers, as well as any fibers adhering to metallic clips or staples. A second high density cleaner removes the metal. A vibrating screen removes contaminants such as foil, plastic, latex particles, and low-specific-gravity material. The stock is then fully delaked and stored in a chest until any bleach added is exhausted. Storage longer than 2 hr will darken the stock because ink particles will redeposit on the fibers or will produce ink cloots that are difficult to remove. Low-density cleaners may also be used, some with core bleed to remove all small contaminants. The stock is pumped to a distribution box, the consistency regulated to 0.8 to 1.2%, depending on the quality desired, and the stock then metered into the flotation system. A constant flow and consistency required for flotation.

The air bubbles generated in the flotation cell are stabilized by the foaming

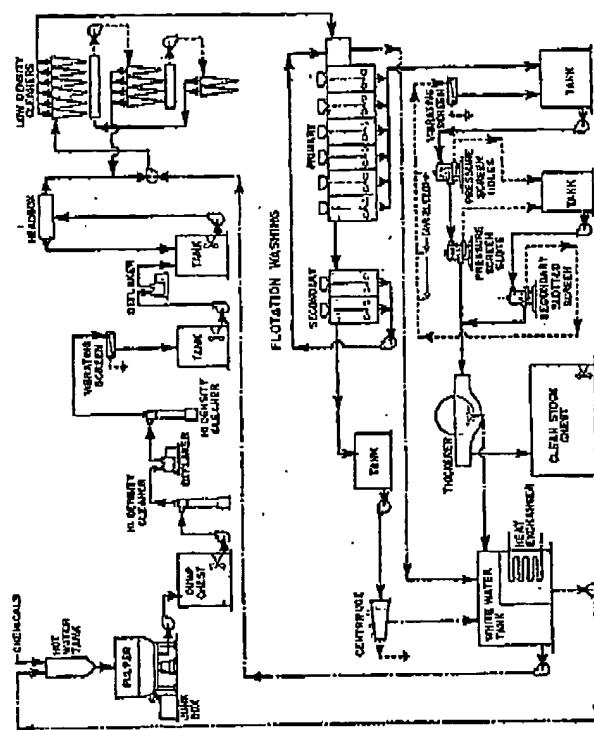


Figure 4-132. Flotation deinking and washing system.

chemicals through a decrease in the surface tension of the water. Although the pigments are heavier than water, they remain suspended because of their small size and hydrophobic properties. The collector chemical attaches the ink particles to the air bubbles so they can be floated to the surface to form a layer of foam that is directed toward the skimmer for removal. The amount and type of foamer and collector chemicals are selected to provide the desired buoyancy. If the exact ratio is not used, the performance can be greatly affected. The floated stock is then screened through holes of 0.16 to 0.20 cm (0.062 to 0.079 in.) in diameter, followed by screening through slots of 0.010 to 0.018 in. and then thickened. A complete flow diagram of a flotation deinking and washing system is shown in Figure 4-132. Horacek¹⁸ⁿ has pointed out that dispersed ink particles in the range of 8 to 10 μ m can be removed by press washing at high consistency (up to 35%) which reduces the effluent that needs to be processed and/or treated.

Shrinkage of Wastepaper in Deinking

Shrinkage, which is the ratio of the total loss to the total furnish, is an important economic factor in deinking of wastepaper. Among the factors that affect shrinkage are the composition of the wastepaper, losses caused by mechanical

592 PULPING treatment, and losses due to chemical treatment. Shrinkage on washing varies from about 8 to 10% for ledger stock, to 20 to 35% for book and magazine stock. The total loss of stock in drinking usually ranges from 15 to 40%. Heavily coated book paper when used alone may result in as much as 40 to 60% shrinkage. Fiber loss is relatively low when long-fibered ledger stock is used, but is very high when short-fibered, pigment-coated book papers are used. The data needed to calculate shrinkage, all data being on a moisture-free basis, are as follows:

1. Weight of paper furnished, including rejects (A).
2. Weight of material in water furnished to the pulper (B).
3. Weight of chemicals added (C).
4. The total loss, including all sorting rejects, soluble, and insoluble matter in the plant effluent (D).

The percent shrinkage is then calculated by the formula:

$$\frac{D}{A+B+C} \times 100$$

The relative pulp yield for various grades of wastepaper are shown below.¹¹⁷ High ash content in the wastepaper is particularly important in its effect on the yield.

Type of Paper	Ash (%)	Yield (%)
Bond	2	90
Ledger	5	85
Offset	12	81
Book	20	66
Coated	25	58
Coated	30	50

Bleaching of Delinked Stock

Well-washed delinked stock ranges in color from a fairly bright, blue-white for ledger stock to a dull gray when mixed papers are used. The brightness may be as high as 60 GE. As mentioned above, peroxide may be used in the cooking operation to improve the brightness of the pulp, particularly when groundwood papers are present. Peroxide may also be used as a bleaching agent after cooking using about (1) 3.0% sodium hydroxide and 1.25% hydrogen peroxide or (2) 1.5% sodium hydroxide and 1.5% sodium peroxide. If brightness in the 80 plus range is desired when delinking chemical fibers, a bleaching stage using hypochlorite can be used. There are three common systems, which are described below.

SECONDARY FIBER PULPING 593

Single-stage Sodium Hypochlorite. Thickened stock from the washers is watered with an inclined-screw thickener to 20% consistency, mixed with 0.8% chlorine as sodium hypochlorite, heated to 50 to 60°C, and held for a retention time of 60 min. Sometimes a lesser amount of chlorine is used, especially if the bleached stock is to be used in low-grade napkin or toweling.

Chlorination Plus Hypochlorite. The thickened stock at 4 to 5% consistency is treated with chlorine gas and pumped through an upflower at a rate to provide a 15-min retention time, and then sodium hydroxide is added to make a hypochlorite stage. A retention time of 60 min is provided. The disadvantage of this system is the low consistency required, the relatively high losses in the chlorine stage, and the low degree of effectiveness in the hypochlorite stage. Normally a 7 point brightness increase is the best that can be attained.

Three-stage Bleach System. This consists of direct chlorination in a tower, caustic extraction in a tower, caustic washing, and finally a hypochlorite stage. The brightness increase using this method is usually 10 to 11 points. A bleaching process in which the hypochlorite is added to thickened stock before washing is described below. After pulping with chemicals, the stock is dewatered to 25% consistency and simultaneously heated to 49 to 60°C. The pressed-out chemicals and hot water are stored for reuse. The chemicals, especially sodium hydroxide, are only about 15% consumed in the pulping process, and, therefore, the cost of chemicals is reduced considerably by this action. The thickened stock is mixed with enough hypochlorite bleaching liquor to bring it to the desired brightness after washing. The degree of brightness obtained is directly related to the amount of chlorine added, as shown in Figure 4-133, where brightness is plotted against chlorine used in a delinked stock made from chemical papers. The amount of bleach and the cost of bleach in this manner is not substantially different from that of a conventional

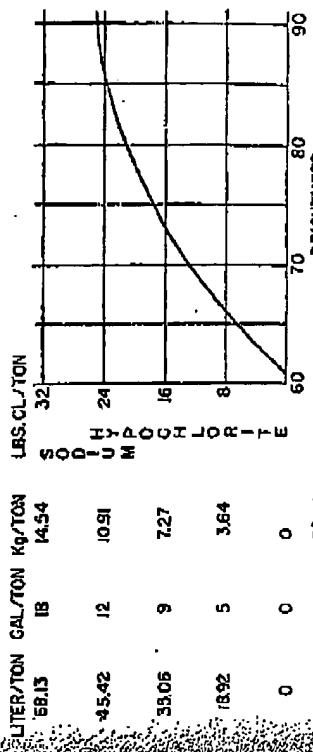


Figure 4-133. Brightness obtainable in bleaching process when hypochlorite is added to thickened stock at 25% consistency.

PULPING OF RAGS AND COTTON LINTERS 595

system. Although it is unorthodox to add bleach liquor immediately after dewatering, while the caustic, ink, and pigments are still present, the method has proven successful. This *aliquot* system is based on the fact that at pH 8 the chlorine absorption is small and the ink, being inert, will not absorb chlorine. The remaining color will be the same as in a conventional system. Using this system, newsprint can be deinked and processed into a good groundwood substitute. With this process the newsprint is pulped with selected chemicals at a temperature of 54°C, subjected to defibering for 45 min, dumped into a storage chest, dewatered to not more than 20% consistency, bleaching chemical added, the stock rediluted in a tower, and the stock then cleaned, screened, and washed.

Bleached deinked stock has a tendency to revert in color. Reversion depends on the conditions used in cooking (caustic soda being worse than other deinking agents), as well as the conditions of bleaching.¹⁷⁴ High pH in hypochlorite bleaching reduces reversion. The use of peroxide for cooking or bleaching is also helpful. High temperatures up to 71°C in hypochlorite bleaching help to reduce reversion.

Properties of Deinked Stock

Most deinked stock has a slightly grayish cast because of the small amount of carbon retained by the fibers. The stock made from chemical papers is used for much the same purposes as soda pulp and is generally competitive with soda pulp in price. It is used in book papers and in ceiling rawstock, where it improves the bulk, opacity, softness, and formation of the paper. The pulp requires no heating. In fact, heating injures the stock, and, for this reason, deinked stock should always be added after the refining when used in mixed furnishes. Ordinarily, the final ash content of deinked stock is in the range of 2.5 to 4.0%. Even excessive washing will not reduce the ash content below about 1.0%.¹⁷⁵ The pH is generally around 8.0 to 8.5.

of the total raw material used for papermaking because of their limited supply and high cost compared with wood.

Use of Rags for Papermaking

High-grade cotton and, to some extent, linen rags are used to make the best grades of bond, writing, and technical papers, where permanence, high strength, and distinctive quality are of interest. Low-grade rags are used for the manufacture of felts used as a base for floor coverings and roofbase materials. In addition to rags, the paper industry utilizes other fiber wastes, such as rope, twine, tent material, and burlap for making strong wrapping papers. This material is generally sold as hard waste fiber, which may contain jute, sisal, manila, caro, or hemp fibers. The availability of this material varies quite widely and depends on market condition.

Selection of Rags

Practically no old rags are used for high-grade paper that must have extreme cleanliness, permanence, and extra high-strength properties. The major source of rag fiber is waste from textile-mill weaving or from garment manufacturers. The selection of rags suitable for high-grade papers has become exceedingly difficult due to contamination from synthetic fibers, such as nylon, cellulose acetate, and polyester. Rayon is also classified as a synthetic fiber by rag-pulp producers in relation to cotton. Fiber cloth clippings containing 100% synthetic fiber are easily detected by expedited rag sorters, but synthetic fibers blended with cotton in the weaving processes are sometimes difficult to detect and are unusable. The increased number of dyes that cannot be stripped without severe damage to the fiber further reduces the available fiber for the cotton-pulp producer. Wool or wool-cotton mixtures cannot be used to make pulp because the alkaline cooking condition used in pulping destroys the wool. Another situation making the selection of rags that are suitable for papermaking extremely difficult has been the increase in the use of synthetic finishes for permanent press and water repellency. These materials, such as vinyl resins, acrylates, urea-formaldehyde resins, and latexes cannot easily be removed by the usual rag processing methods. These materials, when removed from the rag fiber, tend to agglomerate and escape the centrifugal cleaners and later plug the felts and wires and appear in the paper as shiny spots especially after machine calendering.

Rag processing methods. These materials, when removed from the rag fiber, tend to agglomerate and escape the centrifugal cleaners and later plug the felts and wires. Although some cotton waste is produced directly from garment manufacturers or textile mills, it is usually procured from a dealer. Dealers frequently contract for the entire waste from a factory and then sort it into cotton, wool, blends, or straight synthetic. The cotton is further sorted into categories, such as unbleached muslin, white shirt, underwear, slacks, trousers, bleachable

594 PULPING


[my account](#) [learning center](#) [patent cart](#) [document cart](#) [log off](#)
[home](#)[research](#)[patents](#)[alerts](#)[documents](#)**CHAT LIVE**

Be Back Shortly!



Mon-Fri 4AM to 12AM ET

Your Results

- [Tech Track](#)
- [Research Report](#)
- [Retro Results](#)
- [TOC Journal](#)

[My Folders](#) [\[add\]](#) [\[edit\]](#)[Trash \(0\)](#)**RESEARCH REPORT**[Rate This Report](#) [Back to Results](#)

47 of 48

[Previous](#) [Next](#)**Chlor-alkali prices: caustic rollercoaster.**

10-10 TCD1010008602 NDN-094-0007-363B-2 Elsevier Elsevier

journal name-Chem. Ind. (London)
 abbreviated journal title-*clond*
 number 4
 publication date-94-02-21
 PP 132
 document type-Journal, Column
 ISSN-00093068
 CODEN-CHINAG
 language-English

Since chlorine and caustic soda are made together in nearly equal amounts, their prices traditionally waxed and wained in opposing cycles. In general, when caustic soda was in demand, its price rose, but the extra chlorine produced was not wanted, so its price fell, and vice versa. However, the constancy of caustic prices in 1987 was believed to mark a fundamental change in this cycle and the greater demand for caustic was expected to control prices more than chlorine from then onwards (chlorine has fallen out of favour on environmental grounds). The theory has been thrown into confusion by the fact that caustic prices have dropped sharply over the past 2 years. Spot prices for caustic soda in Western Europe have fallen to around \$ 50/tonne from about \$ 400/tonne in 1988-1991. Chlorine demand in Western Europe has also fallen from a peak of 10,000 tonnes/y in 1989 to only 8500 tonnes/y now. The fall in demand for caustic soda has been attributed to reduced inter-regional deep-sea trade and to the fall in demand for caustic from alumina producers. Spot prices for caustic soda are expected to move sharply upwards in 1995 when the alumina market has settled down and to continue to rise up to the year 2000.

[Send More Like This](#)Click here to be [sent more results like this](#)[Add To Folder](#)[Copy to folder...](#) [OK](#)[Get Item](#)[Get this item](#)

descriptor-chemical businesses generally; trends - general
 general industrial code-MS-00; TR-40

cas substance name-chlorine
 sic code-2812
 cas registry number-7782-50-5
 country-Western Europe
 country code-11000
 business term-market
 fact date-1989-1994

cas substance name-caustic soda
 sic code-2812
 cas registry number-1310-73-2
 country-Western Europe
 country code-11000
 business term-pricing
 fact date-1988-1994

**This Page is Inserted by IFW Indexing and Scanning
Operations and is not part of the Official Record**

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

BLACK BORDERS

IMAGE CUT OFF AT TOP, BOTTOM OR SIDES

FADED TEXT OR DRAWING

BLURRED OR ILLEGIBLE TEXT OR DRAWING

SKEWED/SLANTED IMAGES

COLOR OR BLACK AND WHITE PHOTOGRAPHS

GRAY SCALE DOCUMENTS

LINES OR MARKS ON ORIGINAL DOCUMENT

REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY

OTHER: _____

IMAGES ARE BEST AVAILABLE COPY.

As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.